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Modification of silicon nanowires with silver nanoparticles for gas sensor applications

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Abstract. This study focuses on methods to modify the adsorption properties of silicon nanowires produced through plasma cryogenic etching. This research demonstrates the potential for synthesizing a nanocomposite composed of silicon nanowires and silver nanoparticles, which can be utilized to develop highly efficient gas sensors.

Keywords: nanowires, selective adsorption sensor, electrical impedance spectroscopy

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Модификация нитевидных нанокристаллов кремния наночастицами серебра для решения задач газовой сенсорики

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Аннотация. Работа посвящена разработке высокочувствительных газовых сенсоров на основе нитевидных нанокристаллов кремния (ННК). Сенсоры были использованы для детектирования биологических маркеров здоровья человека на примере аммиака и соляной кислоты.

Ключевые слова: кремний, серебро, ННК, наночастицы, сенсорика

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Introduction

The influence of analyte adsorption on the electronic properties of nanostructures has been extensively utilized in sensor technologies, including those based on optical, resistive, capacitive, and current-voltage analyses. Among the earliest materials employed for gas detection were polycrystalline metal oxide films, such as SnO_2 [1, 2] and ZnO [3, 4], which were fabricated in various configurations [5]. The electronic behavior of these sensors is primarily determined by surface depletion effects caused by adsorption processes. Classical materials such as silicon and III-V compounds are also actively used to form sensor devices. The synthesis of these materials can be carried out both by epitaxial methods ("bottom-up") [6] and, for example, by etching methods ("top-down") [7]. As a result, it becomes possible to synthesize nanostructures of complex morphology, which makes it possible to increase the sensitivity of gas sensors [7]. However, such sensors often suffer from significant limitations, including reduced performance in humid conditions and limited selectivity. In this study, we develop gas sensors based on silicon nanowires (Si NWs) and systematically investigate their electronic properties using electrochemical impedance spectroscopy (EIS) [7] when exposed to ammonia (NH_3) and hydrochloric acid (HCl) vapors.

Materials and Methods

Top-down cryogen plasma chemical etching of the [001]-oriented B-doped silicon substrate with resistivity of 12 Ω·cm was used for Si NWs vertical array fabrication by Oxford PlasmaLab System 100 ICP 380 (Oxford instruments, UK) according to the protocol reported previously [7]. These nanowires exhibit a high aspect ratio and offer a significantly large surface area (cylindrical shape, approximately 10 μm in length and 150 nm in diameter). The nanowires were detached from the silicon substrate using ultrasonication in water and transferred onto a sensor platform with pre-patterned gold interdigital electrodes, forming the Si NW-based sensor. The electronic properties of the sensor were characterized using electrochemical impedance spectroscopy (EIS) (with the use of a Z500P impedance meter (Elins, Russia)) under ambient conditions and in the presence of target analytes (ammonia and hydrochloric acid). This study investigated three distinct types of Si NW-based sensors: as-grown Si nanowires, Si nanowires treated with a 10% aqueous hydrofluoric acid (HF) solution for 3 minutes, and Si nanowires decorated with silver nanoparticles (Ag NPs). Spherical silver nanoparticles (~20 nm in diameter) were synthesized via colloidal chemistry, deposited onto the as-grown Si NWs by drop-casting the colloidal solution, and dried under a nitrogen stream to remove residual solvent.

Results and Discussion

The as-grown Si nanowires, Si nanowires treated with HF and Si nanowires decorated with silver nanoparticles (Ag NPs) were studied using scanning electron microscopy (SEM, Zeiss Supra25, Carl Zeiss, Germany) (Fig. 1, *a*) and transmission electron microscopy (TEM, Jeol JEM-2100F, JEOL Ltd, Japan) (Fig. 1, *b*–*d*). The HRTEM (Fig. 1, *e*–*g*) and selected area electron diffraction (SAED) patterns demonstrated high crystalline quality of the Si NWs (Fig. 1, *h*) and Ag NPs (Fig. 1, *i*) without amorphization. The Au–NW contacts for all nanowire based sensors are found to be Schottky-type (Fig. 1, *j*). The fabricated sensors were tested upon exposure to HCl and NH_3 vapors. To provide indirect measurement of the fluid chemical composition, aqueous solutions of NH_3 and HCl were poured into 3 ml pools with a diameter of 4 cm and evaporated naturally at ambient conditions. The sensors were located at a distance of 5.0 cm above the pool (Fig. 1, *k*).

To study the sensitivity of the sensors, interdigital electrodes were connected to the impedance meter and EIS spectra were obtained in the presence of air, reference medium – water vapor,

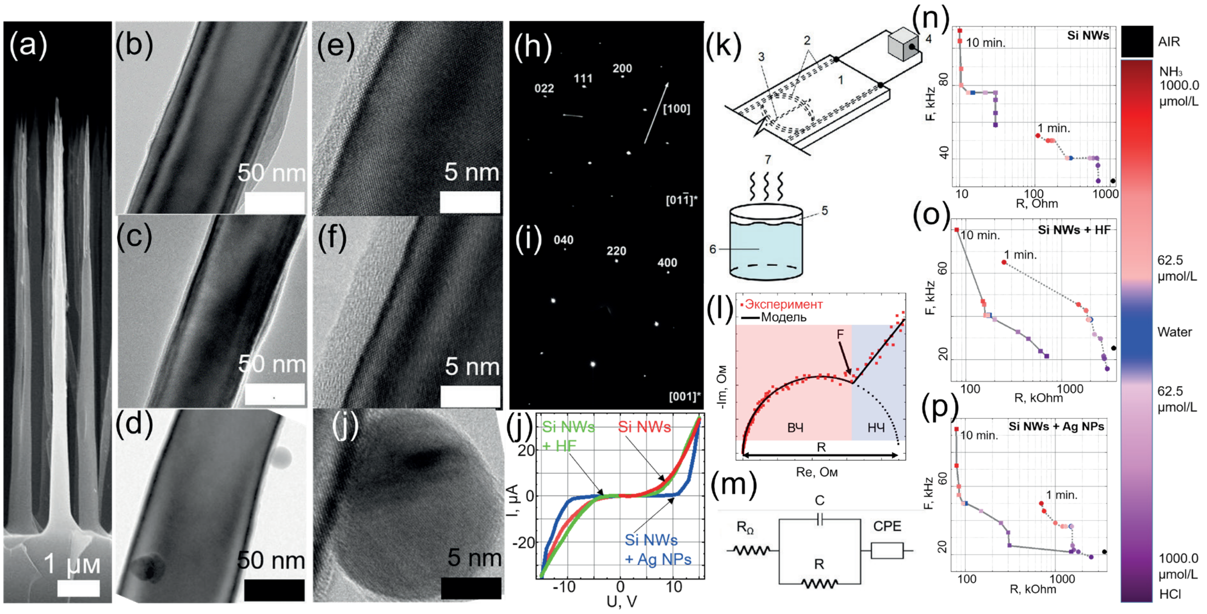

Fig. 1. (*a*) SEM image of the as-fabricated Si NWs. (*b*–*d*) TEM images and (*e*–*g*) HRTEM images of individual as-fabricated Si NW, NW treated with HF and NW decorated with Ag NPs, respectively. (*h*) selective area electron diffraction (SAED) pattern of a Si NW. (*i*) SAED pattern of a Ag NP. (*j*) current–voltage characteristics of the fabricated sensors based on as-fabricated (red), treated with HF (green) and decorated with Ag NPs (blue) Si NWs. (*k*) – Schematic of the measurement setup: 1 – contact platform, 2 – gold electrodes, 3 – NW, 4 – impedance meter, 5 – pool, 6 – solution, 7 – analyzed vapor. (*l*) – typical EIS spectrum of a sensor. (*m*) – equivalent circuit: R_Ω – resistance of the electrodes, *R* – resistance of the sensor, *C* – capacitance of the sensor and constant phase element (CPE). (*n*–*p*) – *F*(*R*) maps for the sensors based on: Si NWs, NWs treated with HF and decorated with Ag NPs, respectively, exposure time 1 min – round dots, 10 min – square dots

and vapors of NH_3 and HCl aqueous solutions in a wide concentration range of 62.5 (1.25 ppm for NH_3 and 1.88 ppm for HCl) to 1000.0 μmol·l^{-1} (20 ppm for NH_3 and 30 ppm for HCl). The impedance spectra were depicted as the Nyquist plots, typical spectrum is presented in Fig. 1, *l*. The obtained curves consist of high-frequency (100 kHz to 500 kHz) and low-frequency (<100 kHz) domains. The high-frequency domain corresponds to the impedance of the NWs and Schottky barrier and represented by a semicircle. The low frequency domain follows nearly linear dependence and is considered as the result of the diffusion processes at the nanowire-gold interface [7]. All the obtained EIS spectra were fitted using the equivalent electrical circuit method. The employed circuit (Fig.e 1, *m*) contains: R_Ω – contact resistance corresponding to the resistance of the gold interdigital electrodes, *R* – resistance related to the Si NWs and Schottky barriers (referred to as the sensor resistance), *C* – capacitance of the sensor and *CPE* – constant phase element associated with the linear low frequency part of the spectra. These parameters evolve with adsorption of the analyte species on the NW sidewalls, so their analysis allows us to quantify the sensor response with the change in the resistive and capacitive characteristics. The carried out fitting allows one to obtain two parameters in the presence of various vapors: *R* and characteristic EIS frequency *F* (see Fig. 2, *l*–*m*) corresponding to the transition between the predominant action of the contact processes described by *CPE* at low frequencies, to the major role of the resistance *R* and capacitance *C* of the NWs and Schottky barriers at higher frequencies. According to the data analysis results, the *R* and *F* parameters are the fingerprint of the atmosphere surrounding the sensor due to the corresponding change in the spectra governed by the analyte species adsorption. So, exposure under NH_3 and HCl vapors of different concentrations can be quantified via *F*(*R*) mapping of the sensory response in order to obtain selectivity in sensing (Fig. 1, *n*–*p*).

Conclusion

It is shown that sensors based on silicon nanowires and silver nanoparticles exhibit an enhanced response to hydrochloric acid vapors compared to sensors made from as-grown silicon nanowires.

In opposite untreated silicon nanowires demonstrated good sensitivity to ammonia vapors. The results of this study are highly promising for the development of advanced semiconductor devices for portable health monitoring applications.

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